

**REMARKS/ARGUMENTS**

No new matter has been added. The amendments to the specification address typographical, grammatical, and spelling errors. The amendments to the claims remove typographical and spelling errors and improve antecedent basis. Therefore, the amendments do not affect, or surrender, any scope of any claim as originally filed.

The Office Action mailed February 12, 2003, has been received and reviewed. Claims 1-12 are currently pending in the application. Claims 1-12 stand rejected. Applicant has amended claims 1-12, added new claims 13-18, and respectfully requests reconsideration of the application as amended herein.

**35 U.S.C. § 112 Claim Rejections**

Claims 1-12 stand rejected under 35 U.S.C. § 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which Applicant regards as the invention. Applicant respectfully traverses this rejection, as hereinafter set forth.

The Examiner alleges that it is unclear whether the reinforcement is carbonized or whether the reinforcement is a preformed layer prior to application of the resin matrix. To clarify the claims, Applicant has amended independent claims 1 and 7 to remove the phrase “as a precursor prior to carbonization.” Therefore, Applicant requests that the rejections under 35 U.S.C. § 112 be withdrawn.

The Examiner also asserts that claims 2, 3, 8, and 9 are unclear because it is unclear how the terms “fibers” and “filaments” differ. Applicant respectfully submits that the terms “fibers” and “filaments” are adequately described in the specification at paragraphs [0021]-[0024]. As such, the scope of claims 2, 3, 8, and 9 is clear to one of ordinary skill in the art and these rejections under 35 U.S.C. § 112 should be withdrawn.

### 35 U.S.C. § 102(b) Anticipation Rejections

#### Anticipation Rejection Based on U.S. Patent No. 4,504,532 to Herring

Claim 1 stands rejected under 35 U.S.C. § 102(b) as being anticipated by U.S. Patent No. 4,504,532 to Herring ("Herring"). A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference. *Verdegaal Brothers v. Union Oil Co. of California*, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). The identical invention must be shown in as complete detail as is contained in the claim. *Richardson v. Suzuki Motor Co.*, 9 USPQ2d 1913, 1920 (Fed. Cir. 1989). Applicant respectfully traverses this rejection, as hereinafter set forth.

Herring discloses a phenolic insulator used in a blast tube of a rocket motor. The blast tube insulator has inorganic particulates and a fibrous reinforcement dispersed in a cross-linked phenolic resin. The fibrous reinforcement includes carbon fibers, polyaramide pulp, or combinations thereof. The blast tube insulator is prepared by dry blending the inorganic particulates, the fibrous reinforcement, and the phenolic resin and then placing the dry mixture into a mold. The dry mixture is then cured to form the phenolic insulator.

As amended, claim 1 recites a method of insulating or thermally protecting a rocket motor assembly. The method comprises forming a reinforcement structure comprising at least one aromatic polyamide. The reinforcement structure is impregnated with a resin matrix to form a rocket motor ablative material and the rocket motor ablative material is used to line a portion of the rocket motor assembly.

Herring does not expressly or inherently describe each and every element of claim 1 because it does not disclose "forming a reinforcement structure comprising at least one aromatic polyamide" or "impregnating the reinforcement structure with a resin matrix to form a rocket motor ablative material." The blast tube insulator in Herring is formed by mixing all of the dry ingredients and then placing the dry mixture in a mold. Therefore, the polyaramide pulp does not form the reinforcement structure. Since Herring does not disclose forming the reinforcement structure from the aromatic polyamide, it necessarily does not disclose impregnating the reinforcement structure with the resin.

Since Herring does not expressly or inherently describe each and every element of claim 1, Applicant respectfully requests that the anticipation rejection of claim 1 be withdrawn.

### **35 U.S.C. § 103(a) Obviousness Rejections**

#### Obviousness Rejection Based on U.S. Patent No. 3,699,210 to Binning *et al.*

Claims 1-7 stand rejected under 35 U.S.C. §103(a) ("Section 103") as being unpatentable over the admitted prior art in view of U.S. Patent No. 3,699,210 to Binning *et al.* ("Binning").

M.P.E.P. 706.02(j) sets forth the standard for a Section 103 rejection:

To establish a *prima facie* case of obviousness, three basic criteria must be met. First, there must be some suggestion or motivation, either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art reference (or references when combined) must teach or suggest all the claim limitations. The teaching or suggestion to make the claimed combination and the reasonable expectation of success must both be found in the prior art, and not based on applicant's disclosure. *In re Vaeck*, 947 F.2d 488, 20 USPQ2d 1438 (Fed. Cir. 1991).

The Examiner bears the burden of establishing that each of these three criteria is met.

Applicant respectfully submits that the Section 103 rejections of claims 1-7 are improper because the cited references do not teach or suggest all the limitations of the claimed invention or provide a motivation to combine to produce the claimed invention.

Binning discloses a method of carbonizing fibers, such as aromatic polyamide fibers. The fibers are first pretreated by heating at a temperature of 180°C-550°C in an oxygen-containing environment for an amount of time sufficient to blacken the fibers. The blackened fibers are then heated in a laser beam in a non-oxidizing environment at a temperature from 700°C-1200°C for longer than one-tenth of a second.

Binning does not teach or suggest "forming a reinforcement structure comprising at least one aromatic polyamide," as recited in claim 1, because Binning does not disclose that its aromatic polyamide fibers are used to form a reinforcement structure. Rather, the aromatic polyamide fibers in Binning are carbonized and then the carbonized fibers are used in nose cones

or rocket nozzle exhausts. Since Binning does not disclose forming the reinforcement structure, Binning necessarily does not teach or suggest "impregnating the reinforcement structure with a resin matrix to form a rocket motor ablative material."

The cited references also do not provide a motivation to combine their teachings to produce the invention of claim 1. "[T]he mere fact that references can be combined or modified does not render the resultant combination obvious unless the prior art also suggests the desirability of the combination." M.P.E.P. § 2143.01. Moreover, the fact that the references relied upon teach that all aspects of the claimed invention were individually known in the art is not sufficient to establish a *prima facie* case of obviousness without an objective reason to combine the teachings of the references. *Id.* Neither the admitted prior art nor Binning teach or suggest the desirability of forming a reinforcement structure from an aromatic polyamide. While Binning discloses preparing carbonized fibers from aromatic polyamides, any resulting structure would be formed from the carbonized fibers and not the aromatic polyamide. In addition, Binning does not teach or suggest that the carbonized fibers provide thermal and ablative protection.

The Examiner states that "[i]t would have been obvious to one of ordinary skill in the art at the time the invention was made to replace the rayon of the admitted prior art with polyaramid since rayon is no longer available and since Binning prefers polyaramid to rayon. It is noted that the fibers form a flexible layer . . . and thus one in the art would appreciate that these materials were intended to be used [as] insulation." Office Action of February 12, 2003, p. 3-4. This motivation provided by the Examiner is not an objective reason to combine the cited references because the necessary ablative and thermal properties of the rocket motor ablative material are not related to the availability of the rayon material. Furthermore, the fact that the fibers in Binning form a flexible layer does not indicate that a reinforcement structure comprising an aromatic polyamide would have the desired ablative and thermal properties for use as the rocket motor ablative material.

Since the cited references do not teach or suggest all the limitations of claim 1 or provide a motivation to combine, Applicant respectfully submits that the Section 103 rejections are improper and should be withdrawn.

Claims 2-6 are allowable, *inter alia*, as depending on an allowable base claim.

As amended, claim 7 recites a method of insulating or thermally protecting a rocket motor assembly. The method comprises forming a reinforcement structure comprising at least one poly(meta-arylaramid). The reinforcement structure is impregnated with a resin matrix to form a rocket motor ablative material. The rocket motor ablative material is used to line a portion of the rocket motor assembly.

The limitations recited in claim 7 are similar to those in claim 1 and, therefore, claim 7 is allowable for substantially the same reasons discussed above with claim 1. Specifically, the cited references do not teach or suggest "forming a reinforcement structure comprising at least one poly(meta-arylaramid)" or "impregnating the reinforcement structure with a resin matrix to form a rocket motor ablative material." Claim 7 is also allowable because the cited references do not teach or suggest that the reinforcement comprises poly(meta-arylaramid), as acknowledged by the Examiner. In addition, the cited references do not provide a motivation to combine for substantially the same reasons discussed above with claim 1.

Since the cited references do not teach or suggest all the limitations of claim 7 or provide a motivation to combine, Applicant respectfully submits that the Section 103 rejections are improper and should be withdrawn.

Obviousness Rejection Based on U.S. Patent No. 3,576,769 to Hirsch et al.

Claims 7-12 stand rejected under Section 103 as being unpatentable over the admitted prior art and Binning as applied to claim 1 above, and further in view of U.S. Patent No. 3,576,769 to Hirsch *et al.* ("Hirsch"). Applicant respectfully traverses this rejection, as hereinafter set forth.

Hirsch discloses a method of semicarbonizing an aromatic polyamide by exposing the aromatic polyamide to a moderate temperature over an extended time period. To semicarbonize

the aromatic polyamide, the temperature is slowly raised from 25°C to 250°C or 500°C over a time period of 45-60 minutes. Hirsch also discloses that exposing the aromatic polyamide to higher temperatures causes products including the aromatic polyamide to become embrittled and weak. In addition, Hirsch discloses that the products obtained by its process are semicarbonized and distinguishes the properties of its products from those obtained by a carbonizing process.

The cited references do not teach or suggest all the limitations of claim 7. As previously discussed, neither the admitted prior art nor Binning teaches or suggests “forming a reinforcement structure comprising at least one poly(meta-arylaramid)” or “impregnating the reinforcement structure with a resin matrix to form a rocket motor ablative material.” Hirsch does not cure these deficiencies. While Hirsch discloses using poly(meta-arylaramid), Hirsch does not teach or suggest that the poly(meta-arylaramid) is used to form a reinforcement structure. Therefore, Hirsch necessarily does not teach or suggest that the reinforcement structure is impregnated with a resin matrix to form a rocket motor ablative material.

The cited references also do not provide a motivation to combine to produce the invention of claim 7. The Examiner states that “[i]t would have been obvious to one of ordinary skill in the art at the time the invention was made to use any type of polyaramid such as NOMEX as the polyaramid in the admitted prior art and Binning since Binning discloses using polyaramids having phenylenes which are not ortho, since Binning does not indicate only specific polyaramids can be used, and since NOMEX is known in the art as a heat-resistant material.” Office Action of February 12, 2003, p. 5. However, the teachings of Hirsch relate to semicarbonizing the aromatic polyamide fibers. Since Hirsch discloses that carbonizing the aromatic polyamide fibers causes them to become weak and embrittled, Hirsch actually teaches away from combination with the cited references.

Since the cited references do not teach or suggest all the limitations of claim 7 or provide a motivation to combine, Applicant respectfully submits that the Section 103 rejections are improper and should be withdrawn.

Claims 8-12 are allowable, *inter alia*, as depending on an allowable base claim.

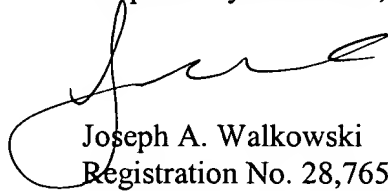
### ENTRY OF AMENDMENTS

The amendments to claims 1-12 above should be entered by the Examiner because the amendments are supported by the as-filed specification. New claims 13-18 are supported by the as-filed specification at paragraphs [0021] through [0025], [0026], and [0027].

### CONCLUSION

Claims 1-18 are believed to be in condition for allowance, and an early notice thereof is respectfully solicited. Should the Examiner determine that additional issues remain which might be resolved by a telephone conference, he is respectfully invited to contact Applicant's undersigned attorney.

Respectfully submitted,



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Date: May 12, 2003

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## **APPENDIX A**

**(CLEAN VERSION OF SUBSTITUTE SPECIFICATION EXCLUDING CLAIMS)**

**(Serial No. 09/896,439)**



**GROUP 1700**

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Attorney Docket 5761.1US

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APPLICATION FOR LETTERS PATENT

for

**ROCKET ASSEMBLY ABLATIVE MATERIALS AND METHOD FOR  
INSULATING OR THERMALLY PROTECTING A ROCKET MOTOR**

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## TITLE OF THE INVENTION

### ROCKET ASSEMBLY ABLATIVE MATERIALS AND METHOD FOR INSULATING OR THERMALLY PROTECTING A ROCKET MOTOR

## CROSS-REFERENCE TO RELATED APPLICATION

**[0001]** The benefit of priority is claimed of U.S. provisional application 60/215,064 filed in the U.S. Patent & Trademark Office on June 30, 2000, the complete disclosure of which is incorporated herein by reference.

## BACKGROUND OF THE INVENTION

**[0002]** Field of the Invention: This invention relates to rocket motor ablative materials, especially resin-filled carbon fiber and carbon/carbon ablative materials, and a method for making the ablative materials. In particular, this invention relates to carbon ablative materials having a reinforcement component formed from, as a precursor prior to carbonization, an aramid material, especially a meta-aramid material. This invention also relates to rocket motor assemblies comprising the carbon ablative materials.

**[0003]** State of the Art: It is generally accepted current industry practice to prepare insulation for solid rocket motors from a polymeric base composite importantly including a carbon cloth. The composite is generally composed of the carbon cloth as a woven reinforcement structure impregnated with a suitable resin matrix. The resin matrix is commonly a phenolic resin, although other resin matrices can be used. For making the woven reinforcement structure, current industry practice is to select a continuous filament non-solvent spun viscose rayon as a precursor material. The continuous filament viscose rayon, which is especially formulated for ablative applications, is woven, wound, or otherwise manipulated into its desired configuration and then carbonized to form a carbon structure exhibiting superior ablation characteristics and excellent physical properties and processability.

**[0004]** Continuous filament viscose rayon precursor has been established as a standard in the rocket motor industry for making carbon reinforced structures of carbon and carbon/carbon ablative materials due to its superb ablative characteristics, excellent physical and thermal properties, and high processability. One of the excellent physical properties possessed by

composites formed from a continuous filament viscose rayon precursor is a cured composite high warp strength of about 144.8 MPa (or about 21,000 lbs/in<sup>2</sup>) at ambient temperature (about 21°C or 70°F), as measured subsequent to carbonization and impregnation of the precursor. Warp strength reflects the tolerance of the filament to opposing forces acting along the warp (or longitudinal) filament axis.

[0005] However, a major drawback associated with the use of cured composites comprising wrapped layers of continuous filament viscose rayon, such as found within the bulk areas of much rocket nozzle insulation, involves the availability of this particular type of continuous filament. Over the past few years, the only manufacturer producing sufficient quantities of continuous filament viscose rayon to meet industry demands is North American Rayon Corp. (NARC) of Elizabethton, Tennessee. The capability of the industry to produce ablative liners and other thermal insulation based on continuous filament viscose rayon has been compromised, however, due to the cessation of continuous filament viscose fiber production by NARC. There is, therefore, a need in this industry, previously not satisfied, to find an effective alternate source or a replacement candidate for the above-described standard thermal insulation formed from a continuous filament viscose rayon precursor.

[0006] The requirements that a replacement candidate must satisfy in order to be acceptable and functionally effective are well known to be quite severe due to the extreme conditions to which the insulation is exposed. These conditions to which the insulation is exposed not only include exceedingly high temperatures but also severe ablative effects from the hot particles (as well as gases) that traverse and exit the rocket motor interior, or pass over the outer surface of reentry vehicle insulators. The insulation must be able to withstand such conditions.

[0007] Accordingly, any replacement insulation should exhibit comparable temperature resistant and ablation characteristics and rheological and physical properties at least equivalent to those of continuous rayon viscose filament, yet should not otherwise significantly alter the manufacturing process employed for the production of the thermal insulation. Additionally, due to the large and growing quantities of solid propellant rocket motor insulation required by the industry, any such replacement reinforcement precursor candidate should be abundantly available now and into the foreseeable future.

[0008] One alternative carbon precursor that has been proposed for ablative applications is continuous filament polyacrylonitrile (PAN). PAN continuous filaments disadvantageously possess higher densities than cellulosic materials ( $1.8 \text{ g/cm}^3$  for PAN, compared to  $1.48 \text{ g/cm}^3$  for cellulosic filaments) and higher thermal conductivities than cellulosic materials. Thus, in order to provide a comparable insulation performance to rayon filaments, rocket motor nozzle insulation or reentry vehicle insulation formed from PAN filament must have a greater thickness and weight than a comparable-performing insulation formed from cellulosic materials. The replacement material must meet the ablation limits for protection of the casing (when used as an internal casing insulation) throughout the propellant burn without adding undue weight to the motor.

[0009] Another alternative carbon precursor is discussed in PCT/US99/18721, which describes an ablative material (*e.g.*, an insulation liner or the like) formed from, as a precursor of a carbon reinforcement structure, yarn comprising carded and yarn-spun cellulosic (*e.g.*, rayon) fibers. The staple cellulosic fibers are processed, such as by spinning, into yarns which, upon patterning (*e.g.*, weaving in any weave style or winding) and subsequent carbonization, serve as a reinforcement. Similarly, PCT/US99/18722 further discloses that a rocket motor ablative material can be formed from, as a precursor of the carbon reinforcement structure, yarn comprising either carded and yarn-spun, solvent-spun cellulosic (*e.g.*, rayon) fibers or solvent-spun cellulosic filaments. Ablatives made from such rayon precursors possess excellent mechanical strength for rocket motor applications, yet do not release unacceptable levels of fiber fly -- *i.e.*, short, waste fibers -- into the air in textile processing operations such as carding, yarn-spinning, and weaving.

[0010] Although staple rayon production is widespread and sufficiently available to those skilled in the art to obviate any obsolescence issues, rayon is relatively time-consuming to produce and expensive due to its low production yields and intensive processing conditions.

[0011] Accordingly, the search for a functionally satisfactory precursor for making the reinforcement structure of a composite material requires discovery and implementation of an extraordinarily complex combination of performance and processing characteristics. Thus, one of the most difficult tasks in the solid propellant rocket motor industry is the development of a suitable, acceptable insulation that will meet and pass a large number of test and processing criteria to lead to its acceptability, yet is relatively inexpensive compared to staple rayon.

## BRIEF SUMMARY OF THE INVENTION

[0012] This invention addresses a crucial need in the industry to reformulate the ablative liners and thermal liners of rocket motors by finding a suitable, inexpensive replacement precursor for making carbon-based reinforcement structures. As referred to above, “suitable replacement” means a precursor material that can be substituted for continuous filament viscose rayon without requiring significant amounts of modification to the impregnating resin composition, component design, and manufacturing process steps. Also, when carbonized and impregnated with a suitable resin, the resulting ablative preferably possesses suitable properties, in particular overall strength, to function in high-temperature environments that a rocket motor is exposed to.

[0013] In accordance with the principles of this invention, these and other advantages are attained by the provision of a rocket motor ablative material (*e.g.*, an insulation liner, bulk material, or the like) formed from, as a precursor of the carbon reinforcement structure, one or more polyarylamides (also referred to herein as aramids or aramides) configured as a suitable reinforcing structure, such as a yarn, flock, and/or felt. Aramid yarns can be prepared by twisting/spinning aramid filaments and/or by carding and yarn-spinning staple aramid fibers. The inventor discovered that aramids are capable of being processed and subsequently carbonized into a prepreg reinforcement that, in combination with a suitable resin matrix, can function as insulation. In particular, the insulation may be used, for example, for a rocket motor nozzle or as a rocket motor heat shield subjected to conditions comparable to those of continuous filament viscose rayon.

[0014] This invention is also directed to a rocket motor assembly comprising ablative materials comprising reinforcing structures formed from, as a precursor prior to carbonization, aramids. This invention is further directed to a process for making a rocket motor assembly comprising the ablative materials, including nozzle and reentry vehicle components.

[0015] Other aspects and advantages of the invention will be apparent to those skilled in the art upon reading the specification and appended claims which, when taken in conjunction with the accompanying drawings, explain the principles of this invention.

## BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0016] The accompanying drawings serve to elucidate the principles of this invention. In such drawings:

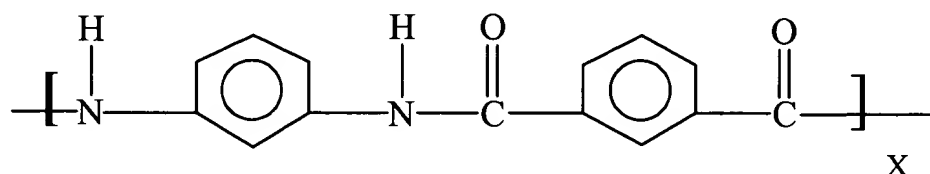
[0017] FIG. 1 is a schematic cross-sectional view depicting the insulation of this invention interposed between a rocket motor casing and solid propellant;

[0018] FIG. 1A is an enlarged view of the insulation of FIG. 1; and

[0019] FIG. 2 is a perspective sectional view identifying some of the regions of a rocket motor nozzle assembly in which the insulation of this invention may be applied.

#### DETAILED DESCRIPTION OF THE INVENTION

[0020] It is presently envisioned to select a poly(meta-arylaramid) and, more preferably, poly(meta-phenyleneisophthalamide) (also known as poly(meta-phenylene isophthalamide)), which is commercially available as NOMEX<sup>®</sup>, as the aromatic polyamide of choice, although this invention may encompass other aromatic polyamides alone or in combination with NOMEX<sup>®</sup>. The aromatic polyamide may also be combined with other suitable precursor materials, such as cellulosic rayon. NOMEX<sup>®</sup> is commercially available from DuPont and has the following structure:



[0021] According to a first embodiment of this invention, the replacement precursor material for preparing carbon reinforcement structures of rocket motor ablative materials comprises bundled aramid filaments, especially aramid filaments twisted/spun into a yarn. The yarn preferably has an average denier per fiber (dpf) in a range of from 1.5 dpf to 3.0 dpf, such as 2.0 dpf.

[0022] In accordance with a second embodiment of the invention, the replacement precursor material for preparing carbon reinforcement structures of rocket motor ablative materials comprises carded and yarn-spun aramid staple fibers. As referred to herein and understood in the art, "carded" means staple fibers subjected to a process or passed through a machine designed to promote the at least partial separation and at least partial alignment of the staple fibers. Carding encompasses techniques used in the production of both fine and coarse yarns. As referred to herein and understood in the art, "yarn-spun" means a yarn formed by a combination of drawing or

drafting and twisting of prepared staple fibers. Spinning (or yarn-spinning) of carded staple fibers as referred to in the context of this second embodiment is not intended to mean techniques consisting of the extrusion of continuous filaments, which techniques can be performed during solvent-spinning. As referred to herein, staple fibers are fibers having lengths suitable for yarn-spinning.

[0023] In the case of aramid staple fibers, the staple fibers preferably have average fiber lengths in a range of from 38 mm to 225 mm, such as 100 mm to 150 mm, and, when processed into a yarn, have an average denier per fiber (dpf) in a range of from 1.5 dpf to 3.0 dpf, such as 2.0 dpf.

[0024] The filaments of the first embodiment and staple fibers of the second embodiment of this invention are preferably untreated, meaning that they are free of any distinct metallic, metalloidal, or graphitic coating, at least prior to (and preferably subsequent to) graphitization.

[0025] One of the advantageous features of this invention is that the yarn comprising either the aramid filaments or the carded and spun aramid staple fibers may be substituted for conventional continuous filament viscose rayon without significantly altering the ablative material manufacturing process. The only substantial alteration from the conventional continuous filament viscose rayon manufacturing process resides in the carding and yarn-spinning of the staple fibers for the second embodiment. Generally, continuous filament viscose rayon is produced by dissolving cellulose into a viscose spinning solution and extruding the solution into a coagulating medium where the polymer is cellulose and is regenerated as a continuous filament. On the other hand, the yarn used in the second embodiment of the present invention is prepared from staple fibers, which are carded and spun into a tight, compact yarn by techniques well known in the industry. It is understood that other processing techniques may also be used, such as combing and other steps well known and practiced in the art. Preferably, the spinning step is performed by either a worsted process or a cotton-ring spinning process. The spinning process is advantageous to keep yarn hairiness to a minimum.

[0026] By way of example, the yarn produced by the first and second embodiments of this invention may have a weight comparable to the weight of standard yarns presently used for carbon ablative materials, i.e., about 1200-2400 denier. This may be accomplished with staple fibers by producing a yarn that is approximately 4.8 English worsted count (Nw) and two-plying the yarn to

obtain the 1200 to 2400 denier configuration, preferably 1600 denier with 800 fibers per yarn. Determination of suitable amounts of twisting is within the purview of those skilled in the art.

[0027] The yarns are then subject to one or more patterning techniques including, by way of example, weaving, winding, and plying into a desired structure. Alternatively, the precursor materials may be subject to a nonwoven process to thereby form, for example, a felt or flock structure. By way of example, the yarn may be woven in a 5 harness satin pattern to provide a fabric width of 152 cm and an area weight of 542 g/m<sup>2</sup>. In this regard, the structuring of the yarns into the desired configurations can be performed in the same manner as that for conventional continuous filament viscose rayon.

[0028] The woven or nonwoven structure is then carbonized to form the reinforcement of the ablative material. Carbonization can take place, by way of example and without limitation, at temperatures of at least 750°C to 2800°C, such as 1250°C or higher. The time/temperature schedule should be selected based on thermal degradation properties of the aramid. Thermographic gravimetric analysis (TGA) can be used to determine scheduling. It is preferable to purge the carbonization chamber with an inert gas, such as argon or nitrogen, which either can be flowed through the chamber or sealed within the purged chamber. Unlike carbonization of conventional PAN fibers, which require oxidative stabilization of the PAN fibers to prevent the PAN fibers from melting, oxidizing agents are unwanted in the carbonization of aramid precursors. This difference greatly reduces the processing time and production expense associated with the preparation of reinforcement based on aramid precursors compared to PAN precursors.

[0029] The carbonized reinforcement structure is then impregnated with an acceptable resin, such as a phenolic resin. A representative phenolic resin is SC1008, available from Borden Chemical of Louisville, Kentucky.

[0030] The inventive ablative and insulation materials can be applied to various parts of a rocket assembly, preferably as multilayered structures. Depending on its intended use, the impregnating resin can be either carbonized or not subject to carbonization prior to application to the rocket motor assembly. For example, the ablative and insulation materials can be used as a chamber internal insulation liner, as shown in FIGS. 1 and 1A. Referring to FIG. 1, the insulation 10, when in a cured state, is disposed on the interior surface of the rocket motor case 12. Typically, a liner 14 is interposed between the insulation 10 and the propellant 16. The

insulation 10 and liner 14 serve to protect the case from the extreme conditions produced by the burning propellant 16. Methods for loading a rocket motor case 12 with an insulation 10, liner 14, and propellant 16 are known to those skilled in the art and can be readily adapted within the skill of the art without undue experimentation to incorporate the insulation of this invention. Liner compositions and methods for applying liners into a rocket motor case are also well known in the art, as exemplified by U.S. Patent No. 5,767,221, the complete disclosure of which is incorporated herein by reference. Although FIG. 1 shows a solid propellant grain, the ablative materials which can be used with other propellant formulations, including hybrid and bi-liquid propellants.

**[0031]** The ablative and insulation materials can also (or alternatively) be applied along the flow path of the nozzle structure through which the combustion products pass, such as is shown by area 20 of the exit nozzle shown in FIG. 2. The ablative materials can be exposed along the flow path and/or can be covered by suitable materials, such as refractory metals.

#### EXAMPLE

**[0032]** The following example illustrates an embodiment that has been made in accordance with the present invention. Also set forth is an example prepared for comparison purposes. The inventive embodiments are not exhaustive or exclusive, but merely representative of the invention.

	Meta-Aramid	Filament Rayon
<b>Yarn Properties</b>		
Yarn Denier (g/9 KM)	1600	1650
Fibers per yarn	800	720
Denier per filament (dpf)	2.0	2.3
<b>Woven Fabric Properties</b>		
Fabric Width (cm)	152	152
Area Weight (g/m <sup>2</sup> )	542	576
Weave Pattern	5 harness satin	8 harness satin
<b>Carbon Fabric Properties</b>		
Fabric Width (cm)	111	109
Area Weight (g/m <sup>2</sup> )	327	271
Carbon Content (%)	95.6	97.7
Carbon Yield (%)	48	22
<b>Prepreg Properties</b>		
Carbon Content (%)	48.7	50.6
Resin Content (%)	35.5	34.2
Filler Content (%)	15.8	15.2
<b>Ablative Properties</b>		
Nozzle Erosion Rate (μm/s)	155	171
Char Depth (mm)	9.4	8.2
Total Heat Effect Depth (mm)	14.7	14.2

[0033] Testing was based upon solid fuel rocket motor test firing of 35 seconds at 6.2 Mpa (900 psi). As seen from the table, the ablative material made from the meta-aramid fiber exhibited properties comparable, if not superior, to those of an ablative material made of filament rayon.

[0034] The foregoing detailed description of the invention has been provided for the purposes of illustration and description. It is not intended to be exhaustive or exclusive in its description of the precise embodiments disclosed. The embodiments were chosen and described in

order to best explain the principles of the invention and its practical application, thereby enabling others skilled in the art to understand the invention for various embodiments and with various modifications covered within the spirit and scope of the appended claims.

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## ABSTRACT OF THE DISCLOSURE

OR A rocket motor ablative material (*e.g.*, an insulation liner, bulk material, or the like) of this invention is formed from, as a precursor of the carbon reinforcement structure, one or more polyarylamides configured as a suitable reinforcing structure, such as a yarn, flock, and/or felt. Aramid yarns can be prepared by twisting/spinning aramid filaments and/or by carding and yarn-spinning staple aramid fibers. In particular, the insulation may be used, for example, for a rocket motor nozzle or as a rocket motor heat shield subjected to conditions comparable to those of continuous filament viscose rayon.

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# **APPENDIX B**

**(VERSION OF SUBSTITUTE SPECIFICATION EXCLUDING CLAIMS  
WITH MARKINGS TO SHOW CHANGES MADE)**

**(Serial No. 09/896,439)**



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APPLICATION FOR LETTERS PATENT

for

**ROCKET ASSEMBLY ABLATIVE MATERIALS, MATERIALS AND METHOD FOR  
INSULATING OR THERMALLY PROTECTING A ROCKET MOTOR**

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## TITLE OF THE INVENTION

### ROCKET ASSEMBLY ABLATIVE MATERIALS, ~~MATERIALS~~ AND METHOD FOR INSULATING OR THERMALLY PROTECTING A ROCKET MOTOR

## CROSS-REFERENCE TO RELATED APPLICATION

[0001] The benefit of priority is claimed of U.S. provisional application 60/215,064 filed in the U.S. Patent & Trademark Office on June 30, 2000, the complete disclosure of which is incorporated herein by reference.

## BACKGROUND OF THE INVENTION

### ~~1. Field of the Invention~~

[0002] Field of the Invention: This invention relates to rocket motor ablative materials, especially resin-filled carbon fiber and carbon/carbon ablative materials, and a method for making the ablative materials. In ~~particular~~particular, this invention relates to carbon ablative materials having a reinforcement component formed from, as a precursor prior to carbonization, an aramid material, especially a meta-aramid material. This invention also relates to rocket motor assemblies comprising the carbon ablative materials.

### ~~2. Description of the Related Art~~

[0003] State of the Art: It is generally accepted current industry practice to prepare insulation for solid rocket motors from a polymeric base composite importantly including a carbon cloth. The composite is generally composed of the carbon cloth as a woven reinforcement structure impregnated with a suitable resin matrix. The resin matrix is commonly a phenolic resin, although other resin matrices can be used. For making the woven reinforcement structure, current industry practice is to select a continuous filament non-solvent spun viscose rayon as a precursor material. The continuous filament viscose rayon, which is especially formulated for ablative applications, is woven, wound, or otherwise manipulated into its desired configuration and then carbonized to form a carbon structure exhibiting superior ablation characteristics and excellent physical properties and processability.

[0004] Continuous filament viscose rayon precursor has been established as a standard in the rocket motor industry for making carbon reinforced structures of carbon and carbon/carbon ablative materials due to its superb ~~ablation~~ablative characteristics, excellent physical and thermal

properties, and high processability. One of the excellent physical properties possessed by composites formed from a continuous filament viscose rayon precursor is a cured composite high warp strength of about 144.8 MPa (or about 21,000 lbs/in<sup>2</sup>) at ambient temperature (about 21°C or 70°F), as measured subsequent to carbonization and impregnation of the precursor. Warp strength reflects the tolerance of the filament to opposing forces acting along the warp (or longitudinal) filament axis.

[0005] However, a major drawback associated with the use of cured composites comprising wrapped layers of continuous filament viscose rayon, such as found within the bulk areas of much rocket nozzle insulation, involves the availability of this particular type of continuous filament. Over the past few years, the only manufacturer producing sufficient quantities of continuous filament viscose rayon to meet industry demands is North American Rayon Corp. (NARC) of Elizabethton, Tennessee. The capability of the industry to produce ablative liners and other thermal insulation based on continuous filament viscose rayon has been compromised, however, due to the cessation of continuous filament viscose fiber production by NARC. There is ~~therefore~~is, therefore, a need in this industry, previously not satisfied, to find an effective alternate source or a replacement candidate for the above-described standard thermal insulation formed from a continuous filament viscose rayon precursor.

[0006] The requirements that a replacement candidate must satisfy in order to be acceptable and functionally effective are well known to be quite severe due to the extreme conditions to which the insulation is exposed. These conditions to which the insulation is exposed not only include exceedingly high temperatures but also severe ablative effects from the hot particles (as well as gases) that traverse and exit the rocket motor interior, or pass over the outer surface of ~~re-entry~~reentry vehicle insulators. The insulation must be able to withstand such conditions.

[0007] Accordingly, any replacement insulation should exhibit comparable temperature resistant and ablation characteristics and rheological and physical properties at least equivalent to those of continuous rayon viscose filament, yet should not otherwise significantly alter the manufacturing process employed for the production of the thermal insulation. Additionally, due to the large and growing quantities of solid propellant rocket motor insulation required by the industry,

any such replacement reinforcement precursor candidate should be abundantly available now and into the foreseeable future.

[0008] One alternative carbon precursor that has been proposed for ablative applications is continuous filament polyacrylonitrile (PAN). PAN continuous filaments disadvantageously possess higher densities than cellulosic materials ( $1.8 \text{ g/cm}^3$  for PAN, compared to  $1.48 \text{ g/cm}^3$  for cellulosic filaments) and higher thermal conductivities than cellulosic materials. Thus, in order to provide a comparable insulation performance to rayon filaments, rocket motor nozzle insulation or ~~re-entry~~ entry vehicle insulation formed from PAN filament must have a greater thickness and weight than a comparable-performing insulation formed from cellulosic materials. The replacement material must meet the ablation limits for protection of the casing (when used as an internal casing insulation) throughout the propellant burn without adding undue weight to the motor.

[0009] Another alternative carbon precursor is discussed in PCT/US99/18721, which describes an ablative material (*e.g.*, an insulation liner or the like) formed from, as a precursor of a carbon reinforcement structure, yarn comprising carded and yarn-spun cellulosic (*e.g.*, rayon) fibers. The staple cellulosic fibers are processed, such as by spinning, into yarns which, upon patterning (*e.g.*, weaving in any weave style or winding) and subsequent carbonization, serve as a reinforcement. Similarly, PCT/US99/18722 further discloses that a rocket motor ablative material can be formed from, as a precursor of the carbon reinforcement structure, yarn comprising either carded and ~~yarn-spun~~ yarn-spun, solvent-spun cellulosic (*e.g.*, rayon) fibers or solvent-spun cellulosic filaments. Ablatives made from such rayon precursors possess excellent mechanical strength for rocket motor applications, yet do not release unacceptable levels of fiber fly -- *i.e.*, short, waste fibers -- into the air in textile processing operations such as carding, yarn-spinning, and weaving.

[0010] Although staple rayon production is widespread and sufficiently available to those skilled in the art to obviate any obsolescence issues, rayon is relatively time-consuming to produce and expensive due to its low production yields and intensive processing conditions.

[0011] Accordingly, the search for a functionally satisfactory precursor for making the reinforcement structure of a composite material requires discovery and implementation of an extraordinarily complex combination of performance and processing characteristics. Thus, one of the most difficult tasks in the solid propellant rocket motor industry is the development of a suitable,

acceptable insulation that will meet and pass a large number of test and processing criteria to lead to its acceptability, yet is relatively inexpensive compared to staple rayon.

### BRIEF SUMMARY OF THE INVENTION

[0012] ~~It is, therefore, an object of this~~ This invention to address addresses a crucial need in the industry to reformulate the ablative liners and thermal liners of rocket motors by finding a suitable, inexpensive replacement precursor for making carbon-based reinforcement structures. As referred to above, ~~suitable replacement~~ “suitable replacement” means a precursor material that can be substituted for continuous filament viscose rayon without requiring significant amounts of modification to the impregnating resin composition, component design, and manufacturing process steps. Also, when carbonized and impregnated with a suitable resin, the resulting ablative preferably possesses suitable properties, in particular overall strength, to function in ~~high temperature~~ high-temperature environments that a rocket motor is exposed to.

[0013] In accordance with the principles of this invention, these and other ~~objects of the invention~~ advantages are attained by the provision of a rocket motor ablative material (*e.g.*, an insulation liner, bulk material, or the like) formed from, as a precursor of the carbon reinforcement structure, one or more polyarylamides (also referred to herein as aramids or aramides) configured as a suitable reinforcing structure, such as a yarn, flock, and/or felt. Aramid yarns can be prepared by twisting/spinning aramid ~~filaments, filaments~~ and/or by carding and yarn-spinning staple aramid fibers. The inventor discovered that aramids are capable of being processed and subsequently carbonized into a prepreg reinforcement that, in combination with a suitable resin matrix, can function as insulation. In particular, the insulation may be used, for example, for a rocket motor nozzle or as a rocket motor heat shield subjected to conditions comparable to those of continuous filament viscose rayon.

[0014] This invention is also directed to a rocket motor assembly comprising ablative materials comprising reinforcing structures formed from, as a precursor prior to carbonization, aramids. This invention is further directed to a process for making a rocket motor assembly comprising the ablative materials, including nozzle and ~~re-entry~~ reentry vehicle components.

[0015] Other ~~objects~~, aspects and advantages of the invention will be apparent to those skilled in the art upon reading the specification and appended claims which, when taken in conjunction with the accompanying drawings, explain the principles of this invention.

#### BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0016] The accompanying drawings serve to elucidate the principles of this invention. In such drawings:

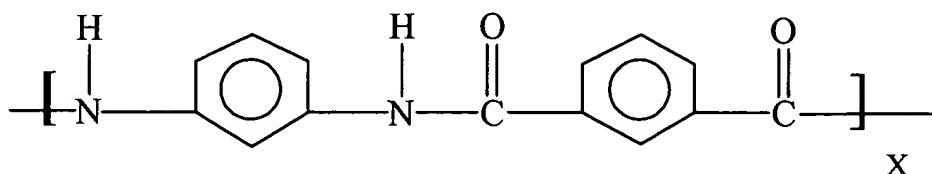
[0017] FIG. 1 is a schematic cross-sectional view depicting the insulation of this invention interposed between a rocket motor casing and solid propellant;

[0018] FIG. 1A is an enlarged view of the insulation of FIG. 1; and

[0019] FIG. 2 is a perspective sectional view identifying some of the regions of a rocket motor nozzle assembly in which the insulation of this invention may be applied.

#### DETAILED DESCRIPTION OF THE INVENTION

[0020] It is presently envisioned to select a ~~poly(meta-arylaramid), and poly(meta-arylaramid) and, more preferably~~preferably, poly(meta-phenyleneisophthalamide) (also known as poly(meta-phenylene isophthalamide)), which is commercially available as NOMEX<sup>®</sup>, as the aromatic polyamide of choice, although this invention may encompass other aromatic polyamides alone or in combination with NOMEX<sup>®</sup>. The aromatic polyamide may also be combined with other suitable precursor materials, such as cellulosic rayon. NOMEX<sup>®</sup> is commercially available from DuPont and has the following structure:



[0021] According to a first embodiment of this invention, the replacement precursor material for preparing carbon reinforcement structures of rocket motor ablative materials comprises bundled aramid filaments, especially aramid filaments twisted/spun into a yarn. The yarn preferably has an average denier per fiber (dpf) in a range of from 1.5 dpf to 3.0 dpf, such as 2.0 dpf.

[0022] In accordance with a second embodiment of the invention, the replacement precursor material for preparing carbon reinforcement structures of rocket motor ablative materials comprises carded and yarn-spun aramid staple fibers. As referred to herein and understood in the art, "carded" means staple fibers subjected to a process or passed through a machine designed to promote the at least partial separation and at least partial alignment of the staple fibers. Carding encompasses techniques used in the production of both fine and coarse yarns. As referred to herein and understood in the art, ~~yarn-spun~~ "yarn-spun" means a yarn formed by a combination of drawing or drafting and twisting of prepared staple fibers. Spinning (or yarn-spinning) of carded staple fibers as referred to in the context of this second embodiment is not intended to mean techniques consisting of the extrusion of continuous filaments, which techniques can be performed during solvent-spinning. As referred to herein, staple fibers are fibers having lengths suitable for yarn-spinning.

[0023] In the case of aramid staple fibers, the staple fibers preferably have average fiber lengths in a range of from 38 mm to 225 mm, such as 100 mm to 150 mm, and, when processed into a yarn, have an average denier per fiber (dpf) in a range of from 1.5 dpf to 3.0 dpf, such as 2.0 dpf.

[0024] The filaments of the first embodiment and staple fibers of the second embodiment of this invention are preferably untreated, meaning that they are free of any distinct metallic, metalloidic, or graphitic coating, at least prior to (and preferably subsequent to) graphitization.

[0025] One of the advantageous features of this invention is that the yarn comprising either the aramid filaments or the carded and spun aramid staple fibers may be substituted for conventional continuous filament viscose rayon without significantly altering the ablative material manufacturing process. The only substantial alteration from the conventional continuous filament viscose rayon manufacturing process resides in the carding and yarn-spinning of the staple fibers for the second embodiment. Generally, continuous filament viscose rayon is produced by dissolving cellulose into a viscose spinning ~~solution~~, solution and extruding the solution into a coagulating medium where the polymer is cellulose and is regenerated as a continuous filament. On the other hand, the yarn used in the second embodiment of the present invention is prepared from staple fibers, which are carded and spun into a tight, compact yarn by techniques well known in the industry ~~into a tight, compact yarn~~. It is understood that other processing techniques may also be used, such as combing and other steps well known and practiced in the art. Preferably, the spinning

step is performed by either a worsted process or a cotton-ring spinning process. The spinning process is advantageous to keep yarn hairiness to a minimum.

[0026] By way of example, the yarn produced by the first and second embodiments of this invention may have a weight comparable to the weight of standard yarns presently used for carbon ablative materials, i.e., about 1200-2400 denier. This may be accomplished with staple fibers by producing a yarn that is approximately 4.8 English worsted count (~~Nw~~), (Nw) and two-plying the yarn to obtain the 1200 to 2400 denier configuration, preferably 1600 denier with 800 fibers per yarn. Determination of suitable amounts of twisting is within the purview of those skilled in the art.

[0027] The yarns are then subject to one or more patterning ~~techniques~~, techniques including, by way of example, weaving, winding, and ~~plying~~, plying into a desired structure. Alternatively, the precursor materials may be subject to a ~~non-woven~~ nonwoven process to thereby form, for example, a felt or flock structure. By way of example, the yarn may be woven in a 5 harness satin pattern to provide a fabric width of 152 cm and an area weight of 542 g/m<sup>2</sup>. In this regard, the structuring of the yarns into the desired configurations can be performed in the same manner as that for conventional continuous filament viscose rayon.

[0028] The woven or ~~non-woven~~ nonwoven structure is then carbonized to form the reinforcement of the ablative material. Carbonization can take place, by way of example and without limitation, at temperatures of at least 750°C to 2800°C, such as 1250°C or higher. The time/temperature schedule should be selected based on thermal degradation properties of the aramid. Thermographic gravimetric analysis (TGA) can be used to determine scheduling. It is preferable to purge the carbonization chamber with an inert gas, such as argon or nitrogen, which either can be flowed through the chamber or sealed within the purged chamber. Unlike carbonization of conventional PAN fibers, which require oxidative stabilization of the PAN fibers to prevent the PAN fibers from melting, oxidizing agents are unwanted in the carbonization of aramid precursors. This difference greatly reduces the processing time and production expense associated with the preparation of reinforcement based on aramid precursors compared to PAN precursors.

[0029] The carbonized reinforcement structure is then impregnated with an acceptable resin, such as a phenolic resin. A representative phenolic resin is SC1008, available from Borden Chemical of Louisville, Kentucky.

[0030] The inventive ablative and insulation materials can be applied to various parts of a rocket assembly, preferably as ~~multi-layered~~multilayered structures. Depending on its intended use, the impregnating resin can be either carbonized or not subject to ~~carbonization~~carbonization prior to application to the rocket motor assembly. For example, the ablative and insulation materials can be used as a chamber internal insulation liner, as shown in FIGS. 1 and 1A. Referring to FIG. 1, the insulation 10, when in a cured state, is disposed on the interior surface of the rocket motor case 12. Typically, a liner 14 is interposed between the insulation 10 and the propellant 16. The insulation 10 and liner 14 serve to protect the case from the extreme conditions produced by the burning propellant 16. Methods for loading a rocket motor case 12 with an insulation 10, liner 14, and propellant 16 are known to those skilled in the ~~art~~art and can be readily adapted within the skill of the art without undue experimentation to incorporate the insulation of this invention. Liner compositions and methods for applying liners into a rocket motor case are also well known in the art, as exemplified by U.S. Patent No. 5,767,221, the complete disclosure of which is incorporated herein by reference. Although FIG. 1 shows a solid propellant grain, the ablative materials ~~of this material~~which can be used with other propellant formulations, including hybrid and bi-liquid propellants.

[0031] The ablative and insulation materials can also (or alternatively) be applied along the flow path of the nozzle structure through which the combustion products pass, such as is shown by ~~the shaded area~~ 20 of the exit nozzle shown in FIG. 2. The ablative materials can be exposed along the flow ~~path~~path and/or can be covered by suitable materials, such as refractory metals.

#### EXAMPLE

[0032] The following example illustrates an embodiment that has been made in accordance with the present invention. Also set forth is a ~~comparative~~comparative example prepared for comparison purposes. The inventive embodiments are not exhaustive or exclusive, but merely representative of the invention.

	Meta-Aramid	Filament Rayon
<b>Yarn Properties</b>		
Yarn Denier (g/9 KM)	1600	1650
Fibers per yarn	800	720
Denier per filament (dpf)	2.0	2.3
<b>Woven Fabric Properties</b>		
Fabric Width (cm)	152	152
Area Weight (g/m <sup>2</sup> )	542	576
Weave Pattern	5 harness satin	8 harness satin
<b>Carbon Fabric Properties</b>		
Fabric Width (cm)	111	109
Area Weight (g/m <sup>2</sup> )	327	271
Carbon Content (%)	95.6	97.7
Carbon Yield (%)	48	22
<b>Prepreg Properties</b>		
Carbon Content (%)	48.7	50.6
Resin Content (%)	35.5	34.2
Filler Content (%)	15.8	15.2
<b>Ablative Properties</b>		
Nozzle Erosion Rate (μm/s)	155	171
Char Depth (mm)	9.4	8.2
Total Heat Effect Depth (mm)	14.7	14.2

[0033] Testing was based upon solid fuel rocket motor test firing of 35 seconds at 6.2 Mpa (900 psi). As seen from the table, the ablative material made from the meta-aramid fiber exhibited properties comparable, if not superior, ~~properties~~ to those of an ablative material made of filament rayon.

[0034] The foregoing detailed description of the invention has been provided for the purposes of illustration and description. It is not intended to be exhaustive or exclusive in its description of the precise embodiments disclosed. The embodiments were chosen and described in

order to best explain the principles of the invention and its practical application, thereby enabling others skilled in the art to understand the invention for various embodiments and with various modifications covered within the spirit and scope of the appended claims.

## ABSTRACT OF THE DISCLOSURE

A rocket motor ablative material (*e.g.*, an insulation liner, bulk material, or the like) of this invention is formed from, as a precursor of the carbon reinforcement structure, one or more polyarylamides configured as a suitable reinforcing structure, such as a yarn, flock, and/or felt. Aramid yarns can be prepared by twisting/spinning aramid ~~filaments~~, filaments and/or by carding and yarn-spinning staple aramid fibers. In particular, the insulation may be used, for example, for a rocket motor nozzle or as a rocket motor heat shield subjected to conditions comparable to those of continuous filament viscose rayon.

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